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Influence of salt added to solvent on extractive distillation

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Abstract

The vapor–liquid equilibria (VLE) of the systems, ethanol–water, ethanol–water–ethylene glycol and ethanol–water–ethylene glycol–$CaCl_2$, at finite concentration and normal pressure were measured. The experimental results showed that ethylene glycol with added salt was more effective than ethylene glycol without salt for separating ethanol and water by extractive distillation. A set of experimental apparatus has been established to measure the relative volatilities of C4 (including butane, 1-butene, 2-*trans*-butene, 2-*cis*-butene and 1,3-butadiene) at infinite dilution by the inert gas stripping and gas chromatography method. It is verified that adding a little salt to the solvents can improve the relative volatilities of C4, and the effect is apparent compared with other organic solvents. Either polar or non-polar systems can be separated by extractive distillation with salt, which is a convenient method for separations to be carried out in industry. © 2002 Elsevier Science B.V. All rights reserved.

Keywords: Extractive distillation; Vapor–liquid equilibria; Ethanol–water; C4; Relative volatilities at infinite dilution

1. Introduction

Extractive distillation is commonly applied in industry, and is becoming a more and more important separation method in petrochemical engineering. It is generally used in two areas [1–5]. One application is separating hydrocarbons with close boiling point such as C4 mixtures, and the other is the separation of mixtures with azeotropic points found with many aqueous ethanol systems.

The two factors influencing the extractive distillation process are the separation step and solvents, that is, separation agents. Assuming that the separation process is determined, the task is to select the basic solvent with high separation ability. When the basic solvent is found, this solvent should be further optimized to improve the separation ability of the solvent and to decrease the solvent ratio and liquid load of the extractive distillation tower.

Extractive distillation with a combination of salt and solvent as the separation agent is a new process for producing high-purity products. This process integrates traditional extractive distillation with the principle of the salt effect. In the application of the extractive distillation process, there is an apparent advantage of the combination of salt and solvent over salt only. In industrial operation, when only salt is used, dissolution, reuse and transport of salt is quite a problem. The concurrent tub-jam and erosion limits the industrial value of extractive distillation with salt only. However, the mixture of solvent and salt can avoid the defects and realize continual production in industry.

In this study, we discuss extractive distillation with salt and solvent combined and select ethanol–water as a representative of polar systems and C4 as a representative of non-polar systems. It is known that both 1,3-butadiene and anhydrous ethanol are basic chemical raw materials. 1,3-Butadiene mainly comes from C4 mixtures and is utilized for the synthesis of polymers on a large scale. Anhydrous ethanol is used not only as a chemical reagent and organic solvent, but also as the raw material of many important chemical products and intermediates. It is reported in the literatures [6–12] that C4 and aqueous ethanol can be separated by extractive distillation. Because these two materials are very important in chemical engineering, their separation by the extractive distillation process is worth studying.

2. Experimental test

A typical recycling vapor–liquid equilibrium (VLE) cell reported in [10], having a volume 100 ml and stirred by magnetic force, was utilized to measure the VLE of the aqueous ethanol system. Salt and the solvent ethylene glycol were blended with the solvent/feed volume ratio 1:1 and the concentration of salt was 0.1 g/ml (salt/solvent). In

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Nomenclature

A	peak area of solutes at intervals of the time t
A_0	original peak area of solutes
D	volumetric rate of the carrier gas (ml/min)
P	saturated vapor pressure (Pa)
R	gas constant ($\mathrm{J\,mol^{-1}\,K^{-1}}$)
S	selectivity
t	interval of the time (min)
T	temperature (K or °C)
x	liquid phase mole fraction
y	vapor phase mole fraction

Greek letters

α	relative volatility
γ	activity coefficient

Subscripts

1	butane
2	1-butene
3	2-*trans*-butene
4	2-*cis*-butene
5	1,3-butadiene
i	component i
j	component j

Superscripts

0	saturated condition
∞	infinite dilution condition

the cell, the mixture with about 50 ml was heated to boiling point at normal pressure. One hour later, the desired phase equilibrium was achieved. At that time, small samples in the vapor and liquid phases were removed by transfer pipet. The samples had a volume 1 ml in order not to destroy the equilibrium. The composition of each sample was determined by local 2305 GC, in which the type of column packing was Porapak Q.

Another set of experimental apparatus have been established to measure the relative volatility of C4 at infinite dilution by the inert gas stripping and gas chromatography method [13,14], which have the merits of a high level of accuracy and reproducibility. A schematic diagram of the experimental apparatus is depicted in Fig. 1. The principle of the method is based on the variation of the vapor phase composition when the highly diluted components of the liquid mixture, controlled to be below 0.01 mol/l, are stripped from the solution by a constant flow of inert nitrogen gas with a flow rate 20 ml/min. In the stripping cell 9, the outlet gas flow is in equilibrium with the liquid phase, and gas is injected into the gas chromatograph 11 by means of a six-way valve 10 at periodic intervals. The peak areas of solutes are recorded by the integrating meter 12. In addition, the method employed by the study involves modifying the construction of the equilibrium cell to enlarge the gas–liquid interface and to increase the contact time between gas and liquid phases by means of a spiral path, and the use of two similar cells, a presaturation cell 8 and a stripping cell 9. The configuration of the stripping cell is illustrated in Fig. 2.

At the beginning of the experiment, both the presaturation cell and the stripping cell are filled with pure solvent such as acetonitrile (ACN) and *N*,*N*-dimethylformamide (DMF). Then the C4 mixture in the vapor phase is admitted from the gas tank into the stripping cell via the liquid and gas inlets. In this way, the quantity of C4 mixture in the solvent is restricted to ensure a high dilution. The necessary data can be obtained by this apparatus in a much shorter time than by other methods [15–19] that are designed to derive the values of the relative volatilities at infinite dilution.

Composition analyses of vapor samples were done by a local gas chromatography (type SQ-206) equipped with a thermal conductivity detector. Sebaconitrile was used as the stationary phase immobilized on the column packing and hydrogen as the carrier gas. The column packing was at room temperature. The relative volatilities of C4's can be

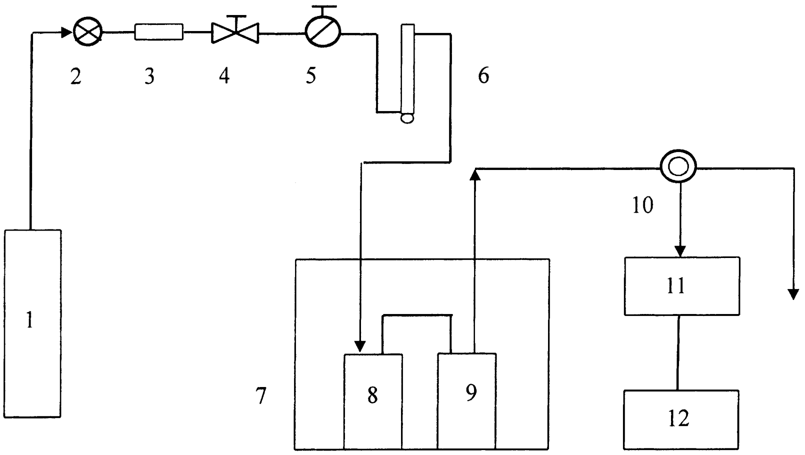

Fig. 1. Schematic diagram of experimental apparatus: (1) gas tank, (2) pressure-reducing valve, (3) gas purifier, (4) pressure-stabilizing valve, (5) current-stabilizing valve, (6) lather flowmeter, (7) thermostatic water bath, (8) presaturation cell, (9) stripping cell, (10) six-way valve, (11) gas chromatograph and (12) integrating meter.

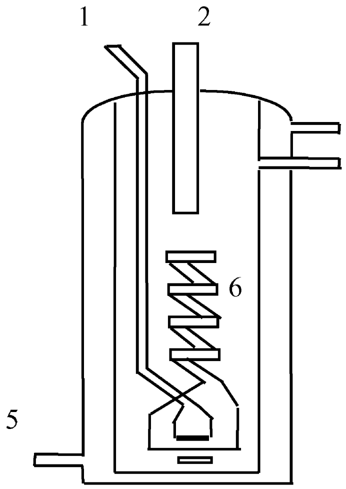

Fig. 2. Configuration of the presaturation and stripping cells: (1) gas inlet, (2) thermometer point, (3) water outlet, (4) gas outlet, (5) water inlet and (6) spiral gas path.

deduced from the change of peak area of vapor samples with time.

The activity coefficient γ_i^∞ of C4 at infinite dilution is given by the following equation:

$$\gamma_i^\infty = \frac{NRT}{DP_i^0 t} \ln \frac{A_0}{A} \qquad (1)$$

where N is the total number of moles of solvent in the dilution cell at time t, and A_0 and A are, respectively, the original peak area of the solute and the peak area at intervals of the time t.

In the extractive distillation process, we usually take the selectivity or relative volatility at infinite dilution as the standard for evaluating the solvent. The selectivity S_{ij}^∞ and the relative volatility α_{ij}^∞ at infinite dilution are, respectively, given by the following equations:

$$S_{ij}^\infty = \frac{\gamma_i^\infty}{\gamma_j^\infty} = \frac{\ln(A_{i0}/A_i)P_j^0}{\ln(A_{j0}/A_j)P_i^0} \qquad (2)$$

$$\alpha_{ij}^\infty = \frac{\gamma_i^\infty P_i^0}{\gamma_j^\infty P_j^0} = \frac{\ln(A_{i0}/A_i)}{\ln(A_{j0}/A_j)} \qquad (3)$$

It is evident that the selectivity S_{ij}^∞ and the relative volatility α_{ij}^∞ are the coordinates in evaluating the separation ability of the solvent. For the sake of simplicity, we select α_{ij}^∞ in this work. Compared with γ_i^∞, the data of α_{ij}^∞ is easy to obtain accurately because it need not measure N and t which may bring some extra errors in the experiment. Thus if the peak area A of the solute at different times is known, α_{ij}^∞ can be calculated according to Eq. (3) in which only the peak area A is required.

3. Results and discussion

3.1. VLE on separating ethanol and water by extractive distillation

First, we measured the equilibrium data of the ethanol(1)–water(2) system which corresponded well with the reference data [20]. It was verified that the experimental apparatus was reliable. Then the measurements were made for the system ethanol(1)–water(2)–ethylene glycol (solvent/feed volume ratio is 1:1) and the system ethanol(1)–water(2)–ethylene glycol–$CaCl_2$ (solvent/feed volume ratio is 1:1 and the concentration of salt is 0.1 g/ml solvent) at normal pressure. The experimental VLE data are listed in Table 1, in which the mole fractions are on a solvent-free basis.

From Table 1, it is shown that under the same liquid composition, the mole fraction of ethanol in the vapor phase with salt is higher than that without salt. It means that adding salt to ethylene glycol is advisable for improving the solvent. Figs. 3 and 4 in which curves are fitted by the Graf4Win software represent the data in Table 1.

Table 1
VLE data at normal pressure, temperature T (K), liquid phase x_1 and vapor phase y_1, mole fraction for all systems; the solvent/feed volume ratio is 1:1 and the concentration of salt is 0.1 g/ml solvent

Systems	T (K)	x_1	y_1
Ethanol(1)–water(2)	372.95	0.0000	0.0000
	372.15	0.0097	0.1035
	368.25	0.0270	0.2248
	364.25	0.0427	0.2967
	362.95	0.0646	0.3612
	359.05	0.1476	0.4870
	356.65	0.2144	0.5475
	354.45	0.3182	0.5810
	353.65	0.4013	0.6137
	353.45	0.5008	0.6485
	353.05	0.5884	0.6915
	352.15	0.6980	0.7520
	351.95	0.7857	0.8049
	351.45	0.8448	0.8515
	351.95	0.9190	0.9158
	351.65	1.0000	1.0000
Ethanol(1)–water(2)–ethylene glycol	424.45	0.0800	0.5190
	421.85	0.1520	0.7160
	417.15	0.5590	0.8130
	416.25	0.7000	0.8725
	415.15	0.8100	0.9085
	414.75	0.9019	0.9490
	414.45	0.9082	0.9570
	413.05	0.9607	0.9790
Ethanol(1)–water(2)–ethylene glycol–$CaCl_2$	425.65	0.0800	0.6790
	423.15	0.1520	0.7900
	419.85	0.5590	0.8820
	418.15	0.7000	0.9170
	416.45	0.8100	0.9449
	415.15	0.9019	0.9672
	414.95	0.9082	0.9690
	414.25	0.9607	0.9860

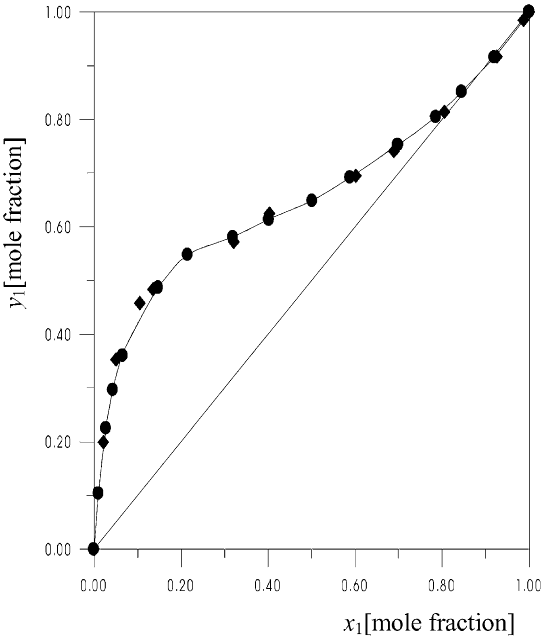

Fig. 3. VLE of ethanol(1)–water(2) at normal pressure. (●) Experimental data; (◆) reference values [20].

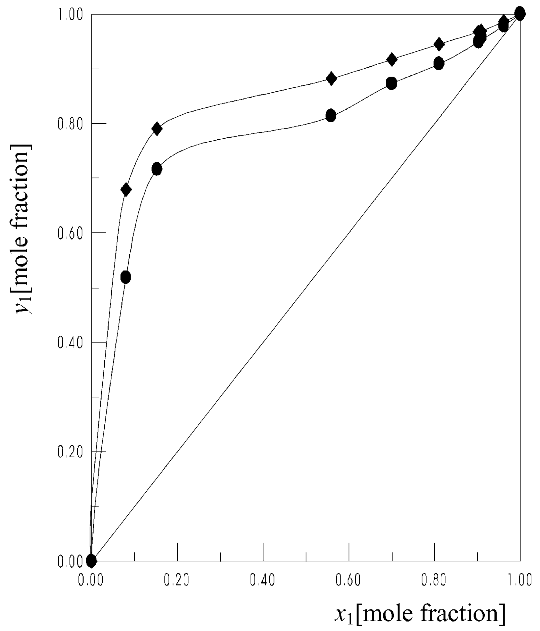

Fig. 4. VLE of ethanol(1)–water(2)–ethylene glycol and ethanol(1)–water(2)–ethylene glycol–$CaCl_2$ at normal pressure. (●) Ethanol(1)–water(2)–ethylene glycol; (◆) ethanol(1)–water(2)–ethylene glycol–$CaCl_2$.

3.2. VLE on separating C4 by extractive distillation

For the C4 system, we measured the relative volatilities of various C4 components at infinite dilution at 20 °C in the solvent ACN and at 50 °C in the solvent DMF. The experimental values are compared with the reference values [21] obtained by the gas chromatography method and are in good agreement except for an error of 12% which may be due to the different methods used to measure the relative volatilities. The results are listed in Table 2, where we use the subscripts 1–5 for butane, 1-butene, 2-*trans*-butene, 2-*cis*-butene and 1,3-butadiene, respectively.

ACN is regarded as a basic solvent; organic solvents, including water, and salts will be added to it. The aim is to find their effect on α_{ij}^{∞} in the solvent ACN. The experimental results are listed in Tables 3 and 4. In Table 3, it is evident that water and ethylenediamine are better additives than the other organic solvents. Table 4 shows that a little salt added to ACN can effectively improve the relative volatilities, and that the effect of the salt is close to that of water and stronger than ethylenediamine. But formerly ACN was mixed with water to separate C4 with the defect that ACN is prone to hydrolyze which thus leads to equipment corrosion and operational difficulties.

DMF is another solvent commonly used to separate C4 mixtures. For the same reason as ACN, DMF is normally used as a single solvent. Many substances are strongly soluble in DMF including many kinds of salts. The influence of salts and organic additives on the separation ability of DMF has been tested. The experimental results are listed in Table 5.

It is seen from Table 5 that the same phenomenon exists as with ACN. Salts added to DMF also improve the relative volatilities of C4 to some degree, and at the same concentration the effect of the salts is more apparent than that of organic solvents. Moreover, if some factors such as relative volatilities, price, erosion, source and so on are considered, the salts NaSCN and KSCN are the best additives.

The influence of NaSCN and KSCN on the relative volatilities at different salt concentrations is also studied. The corresponding relative volatilities and salt composition relationship are diagrammed in Figs. 5 and 6, respectively, for the salt NaSCN and the salt KSCN.

Table 2
The comparison results of experimental and reference values

	ACN (20 °C)			DMF (50 °C)		
	Reference	Experimental	Error (%)	Reference	Experimental	Error (%)
α_{15}^{∞}	3.41	3.82	12.02	3.43	3.32	3.21
α_{25}^{∞}	2.16	2.11	2.31	2.17	2.12	2.30
α_{35}^{∞}	1.70	1.81	6.47	1.76	1.79	1.73
α_{45}^{∞}	1.56	1.63	4.49	1.56	1.61	3.21

Table 3
The experimental results of relative volatilities of C4 at infinite dilution in ACN with organic additives

Organic additives	Weight fraction	T (°C)	α_{15}^{∞}	α_{25}^{∞}	α_{35}^{∞}	α_{45}^{∞}
–	0	28	3.61	2.08	1.77	1.58
2-Butanone	10	28	3.55	2.08	1.75	1.57
2-Butanone	20	29	3.37	2.06	1.71	1.56
Butyl acetate	10	30	3.52	2.04	1.71	1.55
Butyl acetate	20	30	3.30	2.01	1.69	1.53
Ethanol	10	30	3.58	2.09	1.77	1.57
Ethanol	20	30	3.54	2.11	1.77	1.59
Water	10	29	4.06	2.16	1.84	1.62
Ethylenediamine	10	30	3.77	2.11	1.79	1.62
Ethylenediamine	20	30	3.81	2.15	1.83	1.60

Table 4
The experimental results of relative volatilities of C4 at infinite dilution in ACN with salts

Additives	Weight fraction	T (°C)	α_{15}^{∞}	α_{25}^{∞}	α_{35}^{∞}	α_{45}^{∞}
–	0	28	3.61	2.08	1.77	1.58
NaSCN	5	28	3.89	2.15	1.82	1.60
NaSCN	10	29	4.03	2.19	1.85	1.64
KSCN	5	28	3.85	2.14	1.82	1.59
KSCN	Saturated	29	4.00	2.18	1.85	1.64
Polyethylene glycol	5	29	3.64	2.11	1.79	1.59
Polyethylene glycol	10	28	3.74	2.13	1.80	1.61

Table 5
The experimental results of relative volatilities of C4 in DMF with salt and organic additives

Additives	Weight fraction	T (°C)	α_{15}^{∞}	α_{25}^{∞}	α_{35}^{∞}	α_{45}^{∞}
–	0	30	3.92	2.35	1.96	1.69
$AlCl_3 \cdot 6H_2O$	5	25	4.60	2.58	2.12	1.87
$AlCl_3 \cdot 6H_2O$	10	26	4.66	2.62	2.20	1.90
$NaNO_3$	5	30	4.06	2.42	2.02	1.81
$NaNO_3$	10	30	4.32	2.47	2.04	1.82
$CuCl_2 \cdot 2H_2O$	5	28	4.41	2.50	2.05	1.82
$ZnCl_2$	5	28	4.18	2.42	1.99	1.77
$ZnCl_2$	10	28	4.31	2.44	2.03	1.76
NaI	5	30	4.29	2.46	2.00	1.80
KI	5	30	4.25	2.44	2.02	1.76
KI	10	29	4.46	2.51	2.07	1.79
NaSCN	5	30	4.36	2.47	2.05	1.79
NaSCN	10	30	4.53	2.55	2.11	1.85
KSCN	5	31	4.25	2.47	2.04	1.78
KSCN	10	31	4.45	2.57	2.13	1.85
NaBr	5	30	4.11	2.42	2.03	1.80
NaBr	Saturated	30	4.45	2.49	2.07	1.82
Pentaerythritol	5	30	3.94	2.38	1.97	1.76
Pentaerythritol	10	30	4.16	2.43	2.04	1.81
Ethylene glycol	10	30	3.84	2.33	1.95	1.70
Ethylene glycol	20	30	3.91	2.37	1.97	1.72
2-Aminoethanol	10	30	3.93	2.35	1.95	1.70
2-Aminoethanol	20	30	3.88	2.33	1.94	1.71
Ethylenediamine	10	30	3.82	2.32	1.93	1.72
Ethylenediamine	20	30	3.93	2.36	1.96	1.71

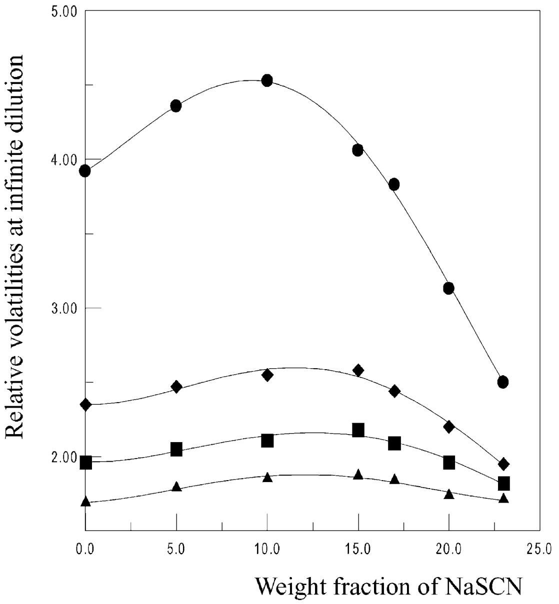

Fig. 5. The relative volatilities of C4 at infinite dilution at different concentrations of NaSCN in DMF at 30 °C: (●) α_{15}^{∞}; (◆) α_{25}^{∞}; (■) α_{35}^{∞}; (▲) α_{45}^{∞}.

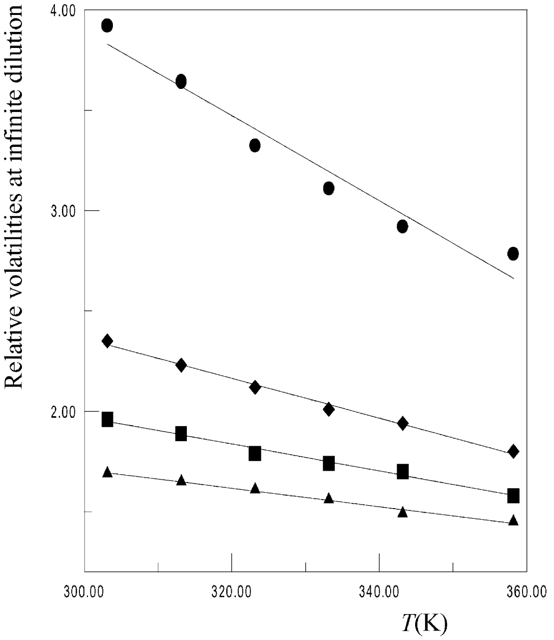

Fig. 7. The relative volatilities of C4 at infinite dilution at different temperatures in solvent DMF: (●) α_{15}^{∞}; (◆) α_{25}^{∞}; (■) α_{35}^{∞}; (▲) α_{45}^{∞}.

The following conclusions are drawn from Figs. 5 and 6:

1. As the salt concentration increases, the relative volatilities of C4 at first increase. But at certain salt concentrations, the relative volatility of C4 can reach a peak and then decrease at different rates. The reason may be that in a solution with high salt concentration, the tendency to ion combination becomes significant, which is unfavorable for making C4 separation because the combined ions are confined and have less chance to make contact with C4 molecules. However, it is known that the electron cloud of 2-*cis*-butene is more mobile than those of butane, 1-butene and 2-*trans*-butene. It means that the attractive force between 2-*cis*-butene and salts is much stronger. Therefore, the relative volatilities α_{45}^{∞} decrease at slow rates for the salt NaSCN and remain almost constant for the salt KSCN.
2. The interaction forces between ions and C4 solutes are somewhat lower than those between ions and the DMF solvent. As a result for C4, the salt effect, salt-out, is brought out. If the salt concentration is very high, there are some unfavorable factors [22] such as low solubility in solvents and decreasing relative volatilities. Therefore, the most suitable concentration of salts NaSCN and KSCN is 5–10%.

On the basis of these findings, we selected 10% NaSCN in DMF as the additive. The relative volatilities of C4 at infinite dilution at different temperatures were measured in DMF and DMF with salt. The experimental results are presented in Figs. 7 and 8.

Whether it is DMF or DMF with salt, the relative volatilities of C4 decrease with increase in temperature, and at the same temperature the relative volatilities of C4 in DMF with added salt are higher than those in DMF without added salt at infinite dilution. So this is an effective way to optimize DMF for C4 extractive distillation. The use of salt, which is convenient, thus has a strong practical value.

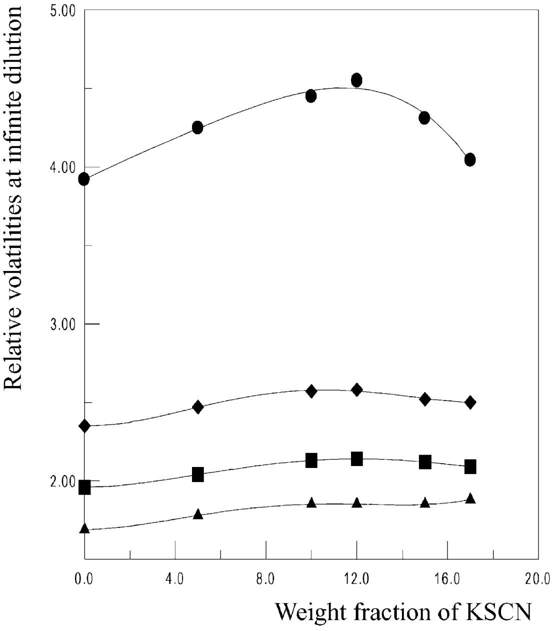

Fig. 6. The relative volatilities of C4 at infinite dilution at different concentrations of KSCN in DMF at 30 °C: (●) α_{15}^{∞}; (◆) α_{25}^{∞}; (■) α_{35}^{∞}; (▲) α_{45}^{∞}.

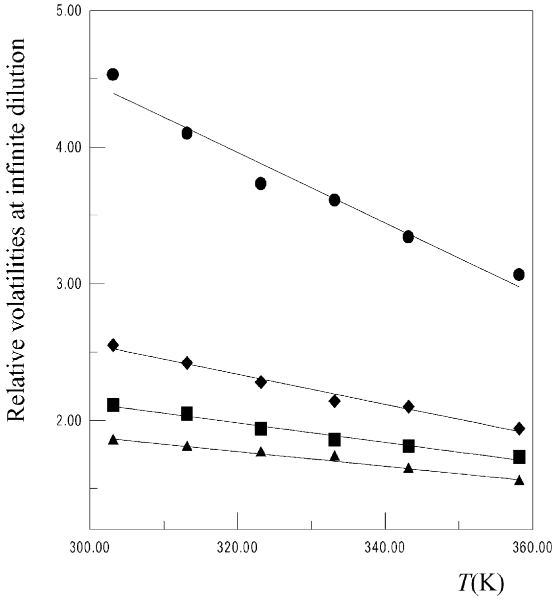

Fig. 8. The relative volatilities of C4 at infinite dilution at different temperatures in DMF with 10% NaSCN: (●) α_{15}^{∞}; (◆) α_{25}^{∞}; (■) α_{35}^{∞}; (▲) α_{45}^{∞}.

In commercial processes, C4 are often in finite concentration in DMF. Because the salt effect of C4 is salt-out, at constant pressure the temperatures of the system DMF/C4 will decrease when salt is added. This will reduce the polymerization of C4 components to some extent, as is desirable in industry.

3.3. *Concluding remarks*

In this work, two sets of experimental apparatuses have been established. One is a typical recycling VLE instrument, which is used for measuring the VLE of ethanol–water, ethanol–water–ethylene glycol and ethanol–water–ethylene glycol–$CaCl_2$ at normal pressure. The experimental results show that ethylene glycol with salt is more effective than ethylene glycol alone for separating ethanol and water by extractive distillation. The salt that we select is calcium chloride ($CaCl_2$) which is easy to get in a common chemical shop.

The other apparatus is the instrument which is used for measuring relative volatilities of C4 at infinite dilution by the inert gas stripping and gas chromatography method. Whether the solvent is ACN or DMF, a little salt added to it can increase the relative volatilities of C4. The effect of salt is more apparent than that of other organic solvents. As the amount of salt rises at low concentration, the relative volatilities of C4 will go up. However, the relative volatilities of C4 will go down as the amount of salt rises at high concentration. The best concentration of salt in the solvent is 5–10%. It is also found that the change in the relative volatilities of C4 is inverse to that of temperature.

The separation process, extractive distillation, is very common in industry. But extractive distillation with salt added to solvent is rarely reported. From our work, we know that in separating either polar or non-polar systems, extractive distillation with salt is still feasible. If we meet problems with extractive distillation in the future, it is worth trying to solve them by adding salt.

It is unfortunate that many salts are corrosive to the equipment and easily decomposed at high temperature. In some cases, the kinds of salts that we can select are limited. The final choice will depend on economic considerations. The benefit from adding salts in the production should exceed the price of salts and other charges. In conclusion, concept of adding a little salt to the solvents to optimize the separation process provides a new method for us.

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